10 Rec'd PCT/PTO 24 SEP 1999

#### -1-

# DEPILATORY COMPOSITIONS, THEIR PREPARATION AND USE

This invention relates to depilatory compositions, in particular those in the form of a gel.

Compositions for removing superfluous body hair are well known. These are of various types. One type of composition is in the form of a cream containing a depilatory agent which degrades hair keratin. In the case of a depilatory cream, hair is removed by applying the cream to an area of skin to be depilated, leaving the cream on the skin for sufficient time for the hair to be chemically dissolved, and then wiping and/or washing the cream and unwanted hair from the skin. Another type of composition is in the form of a wax, which is initially heated before being applied to the skin in a generally molten state. It is then allowed to

Dritish patent specification no. GB 2 223 170

discloses a two-part depilatory composition in which
the first part comprises polyvinyl alcohol (PVA) and
the second part comprises borax and a polyhydroxy
alcohol, or Congo red; the second part functioning to
effect a controlled rate of cross-linking of the PVA.

After in situ cross-linking, such compositions
solidify, requiring the use of a plasticiser to
enable the solid to be peeled from the skin. Such
compositions preferably also include a surfactant.

solidify before being removed from the skin together

with unwanted hair.

Depilatory compositions in the form of a gel are less common and attempts to market such gels have had

15

30

only limited success. The gels tended to have a higher skin irritancy than cream depilatories and became very sticky when being rinsed off because of the nature of the polymer present in the formulation. Moreover, depilatory compositions need to be of high pH, and problems were encountered in formulating a gel which would be stable in such highly alkaline conditions.

It is an aim of the present invention to provide a depilatory composition in the form of a gel which does not suffer from these disadvantages.

It is a further aim of the present invention to provide a depilatory composition in the form of a gel which may be sprayed onto the skin without propellant.

It is another aim of the present invention to provide a depilatory gel having a reduced tendency to irritate the skin.

It has been found that these aims can be achieved by forming a gel from two water-soluble polymeric components and in a pH environment maintained by a preferred buffer system. One of the polymeric components provides the gel with appropriate viscosity and the other polymeric component contributes to the stability of the gel.

Accordingly, one aspect of the present invention provides a depilatory composition in the form of an aqueous alkaline gel comprising:-

- (i) a first polymeric binder comprising a charged, cross-linked, water-soluble polymer;
- (ii) a second polymeric binder comprising a

  linear, water-soluble polymer;
  - (iii) a depilatory agent; and
- (iv) a buffer system capable of maintaining the pH of the composition at a value of from 10.5 to 13.0.

Preferably, the composition remains in the form of an aqueous alkaline gel in use.

- According to another aspect of the present invention, there is provided a process for producing a depilatory composition, which process comprises:-
- (i) forming a first premix by mixing water and a first polymeric binder comprising a charged, cross-linked, water-soluble polymer; forming a second premix by mixing water, a buffer and a second polymeric binder comprising a linear, water-soluble polymer; and optionally forming a third premix by mixing a depilatory agent and a keratin degradation accelerator;
  - (ii) adding a depilatory agent or the third premix to the second premix with stirring;

- (iii) adding the first premix to the resultant
   mixture with stirring; and
- (iv) adjusting the pH to between 10.5 and 13.0.

10

15

20

The depilatory compositions of the present invention accordingly possess unique rheological qualities, which can be defined inter alia by their characteristic viscosity properties. For example, the shear rate (in seconds<sup>-1</sup>) needed to decrease the viscosity of the gel by half is in the range of from about  $10^{-2} \, \text{s}^{-1}$  to about 200  $\text{s}^{-1}$ , preferably from about  $1 \text{ s}^{-1}$  to about  $50 \text{ s}^{-1}$ . Such a large drop in the viscosity of depilatory compositions is not seen with prior art formulations over these shear rate ranges. At a low shear rate of about  $10^{-2} \, \mathrm{s}^{-1}$ , the viscosity of the compositions of the present invention is of the order of  $10^3$  Pas  $(10^6$  cP), whereas at a high shear rate of about 500 s<sup>-1</sup>, their viscosity is of the order of about 0.1 Pas (100 cP). At a very high shear rate of the order of  $4x10^4 s^{-1}$ , such as that effected by a spray-pump (see Table 2 below), it is extremely difficult to measure viscosity, but it can be envisaged that that of the compositions of the present invention would be very low.

25

30

Preferably, a gel in accordance with the present invention has unique viscosity and elasticity, sustained high ionic strength and high pH.

The cross-linked polymer of the first component is such that it swells at high pH (pH 10.5 to 13) and preferably has only carbon-carbon bond

-5-

cross-linkages. More preferably, the elastic modulus of the first component is in the range of from 200 Pa to 1000 Pa, conveniently, 400 Pa to 600 Pa, when measured at a frequency of 10 rad/s; at a temperature of 20°C and a strain of 0.1. The elastic (storage) modulus (G') corresponds to the energy which can be stored and released by the material.

5

10

15

20

30

Suitable first components include a cross-linked acrylic polymer such as a carbomer, for example, Ultrez 10 and/or Carbopol ETD 2020 from Goodrich, and Structure 2000 and 3001 from National Starch.

Another suitable polymer is a cross-linked polyacrylamide such as Seppigel 305. Most preferably, the polymer of the first component is a copolymer of methyl vinyl ether and maleic anhydride cross-linked with dodecadiene available from ISP under the trade designation Antaron Stabilise 06.

Preferably, the depilatory composition includes from 0.1% to 3.0% by weight of the first component.

The second component is a linear non-ionic polymer and/or a positively charged polymer and/or a negatively charged polymer. The second, linear component is one which acts as a suspending agent for the gel and moderates the elastic modulus thereof to enable spreadability. Any suitable linear polymer may be chosen which enables the gel structure to be maintained at the osmotic pressure in the composition. Such linear polymers include hydrophilic polymers having a molecular weight of less than about 600 and non-ionic gel-forming

polymers. For example, the second component may

WO 98/44898

5

10

15

20

25

comprise a water-soluble non-ionic polymer such as polyethylenimine, polyethylene oxide or polyethylene glycol (PEG), a hydrophobically-modified polyol such as Acrysol 44 from Rohm and Haas, or a polyvinyl pyrrolidone such as PVP K from ISP or Luviskol from BASF.

Alternatively, or additionally, the second component may comprise a natural water-soluble polymer such as cellulose, hydroxypropyl cellulose, cetyl hydroxyethyl or propyl cellulose (for example Natrosol Plus from Aqualon), starch, polyglucoside, a polysaccharide such as an alginate, guar gum, carrageenan (lambda, kappa, iota), a sclerotium gum, a polypeptide with or without any alkyl chain (C1 to C18) modification and cationic end groups such as quaternary ammonium groups with counter ions such as chlorides and bromides, or a copolymer of sodium acrylate and dimethyl diallyl ammonium chloride (such as that known as Merquat), or a copolymer of hydroxy ethyl cellulose and dimethyl diallyl ammonium chloride (such as that known as Celquat).

Most preferably, the second component comprises polyethylenimine (such as that known under the trade name Lupasol from BASF) and/or polyvinyl pyrrolidone (such as that known under the trade designation PVP K-30 from ISP).

Preferably, the depilatory composition includes from 0.2% to 3.0% by weight of the second component.

The first and second components, when combined in accordance with the present invention, form a

-7-

polymeric binder having unique viscosity and elasticity.

The depilatory agent may comprise any substance capable of degrading keratin and may be, for example, a sulphur compound such as potassium thioglycolate, dipotassium thioglycolate or thioglycerol.

Preferably, the depilatory composition includes from 0.1% to 6.0% by weight of depilatory agent, preferably from about 3.0% to 5.0%.

10

15

20

Also, the composition may optionally include component(s) which will accelerate the keratin reduction reaction such as urea, thiourea or dithioerythritol, dimethyl isosorbide (DMI), ethoxydiglycol (Transcutol) and methyl propyldiol (MP diol). DMI is preferred.

It is important that the pH of the composition is in the range of from about 10.5 to 13, and preferably about 11.0 to 12.5, to provide good depilation within about 5 minutes. In order to achieve this, a suitable buffer should be present, such as sodium silicate and/or L-arginine, sodium nicotinate and potassium polyethylenimine or a phosphate.

Generally, the buffer system will be present in an amount of from about 2.0% to 8.0% by weight, preferably from about 2.0% to 4.0% by weight.

Advantageously, the buffer system comprises from 2.0% to 3.0% by weight of L-arginine and from 0 to 1.0% by weight of polyethylenimine.

-8-

The pH buffer system preferably ensures efficacy of the depilation within 5 minutes whilst also minimizing skin irritation.

- In order to achieve minimum skin irritation, a composition including L-arginine and/or polyethylenimine, urea and potassium and/or dipotassium thioglycolate is preferred.
- In accordance with a preferred depilatory composition of the present invention, there is provided an aqueous alkaline gel comprising:

15

- (i) 0.1% to 3.0% w/w of a first polymeric binder comprising a charged, cross-linked, water-soluble polymer, preferably a copolymer of methyl vinyl ether and maleic anhydride cross-linked with dodecadiene;
- (ii) 0.2% to 3.0% w/w of a second polymeric binder comprising a linear, water-soluble polymer, preferably polyethylenimine and/or polyvinyl pyrrolidone;
- - (iv) 2.0% to 6.0% w/w, preferably 2.0% to
    4.0% w/w of a buffer system, preferably
    L-arginine and/or polyethylenimine; and,
    optionally,

-9-

(v) 5.0% to 10.0% w/w of an acelerator for the degradation of keratin, preferably urea;

whereby the composition preferably remains in the form of an aqueous alkaline gel in use.

5

10

30

The gels according to the present invention are preferably transparent and remain transparent in use, depending on the amount of active ingredients present and on the procedure of mixing. Generally, they have a thick, rich feel and excellent rinsability with water. Moreover it is not necessary to include a surfactant in the compositions of the present invention, and hence they are less likely to cause skin damage.

15 Due to the special rheology of the polymeric binder, it is possible to include beads, dyes and/or oily particles which will remain dispersed in the Optional excipients include compounds for soothing the skin such as azulene or jojoba and polyethylene glycol ester to reduce irritation. 20 Suitable beads include encapsulated algae extract and sweet almond oil (Elespher). It is also possible to include hydrating, accelerating and/or reducing agents in beads. For example, water-soluble materials such as urea, potassium thioglycolate or 25 thioglycerol can be included in beads, so that these actives are encapsulated.

Although it is possible to provide a gel in accordance with the present invention that has visco-elastic properties suitable for spraying, the depilatory material may alternatively be applied by

means of a roll-on device or in conventional shower gel packaging.

Other ingredients may be included in the compositions of the invention, such as are known in the art.

Unless otherwise stated, all percentage weights referred to herein are % weights based on the weight of the total composition.

10

25

For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, in which:-

- Figure 1 is a graph of shear stress (Pa) against shear rate (s<sup>-1</sup>) for a series of polymeric binders (first component) for the composition of the invention;
- Figure 2 is a graph of the yield points (Pa) of the binders of Figure 1 against the cross-linked polymer (first component) contents (% w/w) of the binders;

Figure 3 is a graph of viscosities (Pas) of the binders of Figure 1 against the shear rate (s<sup>-1</sup>) of the binders; and

Figure 4 is a graph of the elastic (G') and viscous (G'') moduli against stress/strain (Pa) of the first component of the composition.

The following Examples illustrate the invention:

-11-

# EXAMPLES 1 TO 6

A series of depilatory gels was prepared having the compositions shown in the following Table 1:

5 TABLE 1

	11.033	1	2	3	4	5	6
	Phase 1						
10	Polymer A	3.0%	2.5%	2.25%	2.0%	1.5%	1.0%
	deionised water	47.0%	39.0%	36.35%	31.4%	23.5%	15.7%
	Phase 2						
	Polymer B	2.0%	2.0%	2.0%	2.0%	2.0%	2.0%
15	pH buffer	3.0%	3.0%	3.0%	3.0%	3.0%	3.0%
	deionised	10.7%	19.2%	24.8%	26.4%	38.3%	45.8%
	water						
	Phase 3						
	potassium	14.7%	14.7%	14.7%	14.7%	14.7%	14.7%
20	thioglycolate						
	(30%)						
	urea	8.0%	8.0%	8.0%	8.0%	8.0%	8.0%
	Final mix						
25	Phase 1	50.0%	41.5%	37.5%	33.4%	25.0%	16.7%
	Phase 2	15.7%	24.2%	29.8%	33.4%	43.3%	50.8%
	Phase 3	22.7%	22.7%	22.7%	22.7%	22.7%	22.7%
	Potassium	11.6%	11.6%	10.0%	10.5%	9.0%	9.8%
	hydroxide 50%						
	Total	100%	100%	100%	100%	100%	100%
	Total deionised	57.5%	58.0%	59.9%	59.6%	1.6%	61.3%
30	water						

-12-

All amounts are expressed as % weight/weight total composition. Polymer A was a methyl vinyl ether/maleic anhydride copolymer cross-linked with dodecadiene known under the trade designation Antaron ST06 from ISP; and polymer B was polyvinyl pyrrolidone known under the trade designation PVP K-30 from ISP. The pH buffer system was a combination of 2% w/w of L-arginine and 1% w/w of polyethylenimine.

In each case, an Olsa mixer with planar stirring and a high shear stirrer equipped with a vacuum and heating/cooling system was used. The procedure was as follows:

#### Phase 1: Premix polymer A and water.

The water was placed in the mixer and polymer A
was added slowly with stirring. When the polymer had
been dispersed, heating was commenced whilst
maintaining a vacuum of about 0.5 to 0.8 bar to avoid
air bubbles. The vacuum pump was operating
intermittently in order to reduce water loss. When
the temperature had reached about 80°C an almost
clear gel had been formed. This was allowed to cool
and swell overnight.

## Phase 2: Premix polymer B, water, pH buffer

The water and polymer B were introduced into the mixer and homogenised for 2 minutes. The pH buffer system was then added and the mixture homogenised for a further 2 minutes.

-13-

### Phase 3: Urea and potassium thioglycolate

The urea was added to the potassium thioglycolate in a beaker and stirred for 1 hour to complete dissolution.

Final mixing: The final mixing was effected as
follows:-

10

15

20

Phase 2 was placed in the vessel and phase 3 was added with stirring for 15 minutes. A white precipitate appeared. Phase 1 was added while mixing slowly under vacuum (0.5 bar) with planar stirring and the high shear stirrer operated at low speed. The mixture was homogenised for 30 minutes until a lumpy gel had been formed. The potassium hydroxide was then added. The gel became clear and transparent. The pH was adjusted, as necessary, to from 11.0 to 12.5.

Each of the resultant gels was subjected to rheological measurement. The parameters considered were shear stress, shear rate, viscosity and yield point. The results are shown in the Figures.

As can be seen from Figure 1, the variation of shear stress with shear rate is non-linear between 0 and 500 s<sup>-1</sup> and thus the gel has a non-Newtonian behaviour with a yield point. The yield point is the stress required to make the gel flow. Figure 2 shows that the variation of the yield point (TauO), i.e. the shear stress for zero shear rate, varies exponentially. For a small variation in the amount of polymer A, the variation in the yield point, i.e. the variation in spreadability or consistency, may be significant.

-14-

The following table, Table 2, gives characteristic shear rates of products to be sprayed with a conventional pump spray and products to be applied from a shower gel packaging.

# 5 TABLE 2

Type of	Flow Rate (from	Radius of the	Shear rate	
packaging	packaging) of	external output	(s <sup>-1</sup> )	
	product (cm³/s)	(cm)	i	
Pump Spray	0.17	0.0175	500-40000	
		(radius		
		of the		
		actuator)		
Shower gel	8.8	ca 0.15	500-30000	
packaging				
	packaging Pump Spray Shower gel	packaging packaging) of product (cm³/s)  Pump Spray 0.17  Shower gel 8.8	packaging packaging) of external output (cm)  Pump Spray 0.17 0.0175 (radius of the actuator)  Shower gel 8.8 ca 0.15	

The relationship between shear rate and viscosity is shown in Figure 3, from which it can be seen that, at the shear rates given in Table 2, the viscosity is very low.

20

25

30

It has been found that, in order for the gel to be sprayable with a pump spray, its yield point should be not more than from 100 to 200 Pa. If, however, the gel is to be used in a shower gel pack, the yield point may be as high as 300 Pa. Also, in order for the product to remain still for 5 minutes on the skin when held vertical, the yield point needs to be about 60 Pa or more. Thus gels wherein polymer A constitutes from 2.0% to 2.5% w/w are preferred for spraying, whereas rather lower percentages of polymer A may be present where the gel is to be used in the form of a shower gel packaging.

WO 98/44898

5

.10

In a further series of experiments, the blue powder Azulene was dissolved in thick paraffin oil to produce a 1% w/w solution. 2% w/w of this solution was then slowly added to each of the above gels and stirred for 5 minutes. The Azulene remained stably suspended within the gel in the form of blue oily droplets. Depending upon the concentration of Azulene in the premix and the concentration of premix in the gel, the size and colour of the droplets and the overall transparency of the gel could be adjusted.

In a further series of experiments, 0.5% w/w of Elespher was slowly added to each gel and stirred for 5 minutes. Elespher consists of encapsulated beads of algae extract and sweet almond oil, and is green in colour. The beads were stably suspended in the gel.

#### EXAMPLE 7

20

15

Gels were made in accordance with the following Table 3:

TABLE 3

Phase 2	
polymer B	0.6-3
buffer system	3.0
5 water	8.2
Total phase 2	11.8
Phase 3	
water	6.5
potassium	14.7
thioglycolate (30%)	
Total phase 3	29.2
water	to 100.0
КОН 50%	6.15

The amount of water was varied depending upon the amounts of polymers A and B present. Polymers A and B were as used in Examples 1 to 6 and the mixing technique employed was as described in those

Examples. In each case, stable, sprayable depilatory gels were obtained. This showed that for a range of polymer A between 1.0% and 3.0%, a range of polymer B between 0.6% and 3.0% could be used and a stable, sprayable gel formed.